SOP-02
Sample Preservation

Yerington Mine Site
Standard Operating Procedure

Revision 3
Revision Date: May 5, 2008

SOP-02
SAMPLE PRESERVATION

TABLE OF CONTENTS

1.0 OBJECTIVES 1
2.0 APPLICABILITY 1
3.0 RESPONSIBILITY 1
4.0 REQUIRED MATERIALS 1
5.0 DEFINTIONS 2
6.0 METHODS 2
7.0 REFERENCES 4
8.0 ATTACHMENTS 4

1.0 OBJECTIVES

The objective of this standard operating procedure (SOP) is to establish procedures that allow the chemical integrity of a sample is maintained from time of collection until chemical analysis.

2.0 APPLICABILITY

This SOP documents the procedures and chemicals to be used for the preservation of field samples. The environmental media addressed in this SOP include soil, sediment, solid waste, and aqueous samples. These procedures apply to all Project team personnel and subcontractors involved with the collection, shipping and chemical analysis of environmental samples.

3.0 RESPONSIBILITY

The *Project Manager (PM),* or designee, shall ensure that the sampling procedures used, including provisions for proper storage, preservation and shipping, are adequate to maintain sample integrity until custody is assumed by the laboratory. The PM shall develop or direct the preparation of a detailed sampling plan for sampling air, water, biota, sediment, soil, or waste, which shall describe the procedures used to preserve samples during the interval from sampling until receipt by the laboratory.

The *Project Chemist (PC),* or designee, shall ensure that the samples are collected in terms of the analytical methods and compliance with sampling protocols. For smaller projects, the PC and the Field Supervisor may be the same person. The field supervisor or PC also are responsible for maintaining adequate supplies of containers and preservatives. The PM will determine the roles and personnel for each project.

The *Field Supervisor* or his or her designate shall be responsible for ensuring the competence of field sampling personnel and their training. The field supervisor shall ensure that specified preservation and storage procedures are followed during sampling and during shipment to the laboratory.

The *field sampling personnel* will be responsible for the understanding and implementation of this SOP during all field activities. Field personnel are also responsible for checking the collected samples, and verifying that they are preserved with prescribed range.

4.0 REQUIRED MATERIALS

The materials required for this SOP include the following:

- Sample Containers,
- pH instrument or Litmus paper with appropriate pH range,
- Field notebook, and

- Sampling forms (e.g. Chain of Custody Records, sample labels).

5.0 DEFINITIONS

Maximum Holding Time. Maximum Holding Time is the maximum length of time that may elapse before sample preparation (extraction or digestion) or analysis is completed. It is calculated from the date and time of collection in the field. Holding times are usually measured to the nearest day with the exception of those analyses that must be completed within 24 or 48 hours.

Preservation. Preservation refers to temperature control and/or pH adjustment procedures performed to prevent or slow the loss of target analytes through precipitation, volatilization, decomposition, or biodegradation.

Temperature. Temperature is defined as the temperature within the refrigerator, cooler or ice chest that holds the samples. Samples shall be held at 6 degrees Celsius (°C) or less.

6.0 METHODS

Proper communication between the project manager and the analytical laboratory is essential prior to sampling, preferably in writing. This is necessary so that the proper type and number of containers and preservatives can be specified and so that all technical and regulatory requirements can be met regarding the analyses.

Field personnel should coordinate in writing with the laboratory at least two weeks before the sample container kits are to be shipped from the lab to identify the analytes to be requested. The information exchange between lab and field personnel include the project identification, sample kit shipment address, QA/QC regulatory requirements, required turnaround requirements, and the number and type of laboratory analyses.

Most chemical and biological reactions and many physical processes are slowed by lowering the temperature. Therefore, as a general rule, all samples need to be cooled at the time of collection and maintained slightly above freezing until preparation for final analysis. This restriction is not critical in the case of metals analysis since most metals exist in the form of involatile salts with the exception of liquid mercury and organometallic compounds such as tetraethyl lead, which still need to be kept cold. Hexavalent chromium is kept cold to slow its reduction to trivalent chromium.

Soil samples and other solid samples, including sediments, sludges, and solid waste, shall be preserved by cooling to 6° C. Soil and solid samples require no chemical preservatives. However, analysis must be performed within the method-specific holding time requirements.

Aqueous samples may be presumed to be homogenous and amenable to chemical preservation. The following general approaches for chemical preservation shall be employed depending on the analyte(s):

- Volatile acids (HCN, H_2S) are rendered involatile in the presence of strong base (NaOH, pH>12);
- Volatile bases (ammonia) are rendered involatile in the presence of strong acid (H_2SO_4, pH<2);
- Biodegradation of organic compounds is retarded under strongly acidic conditions (HCl or H_2SO_4, pH<2);
- Dehydrohalogenation (loss of HCl) of chlorinated solvents is counteracted in the presence of acid (HCl, pH<2);
- Oxidation of target analytes by the chlorine found in drinking water is eliminated by destroying the chlorine with a reducing agent such as sodium thiosulfate; and
- Many soluble metal salts tend to adhere to the walls of the container or they form precipitates with time. This can be prevented by the addition of nitric acid to a pH of < 2, which maintains the metals as soluble nitrate salts.

Groundwater samples for dissolved metals analysis are filtered (usually with a 0.45 micron filter) before preservation with the appropriate preservative. The filtrate is added directly to the plastic container, which has been supplied with the proper amount of preservative.

With the exception of the stainless-steel sleeves used to capture soil boring samples, all sample containers will be supplied in advance by the subcontracting laboratories.

The required chemical preservatives for aqueous samples will normally be added to the appropriate containers by the subcontracting laboratories before delivery to the field. There are two reasons why already-preserved containers are preferred. First, the laboratory scheduled to perform the analysis maintains control over sample integrity and container cleanliness and, second, field crews are generally not equipped to "appropriately handle" hazardous chemicals like hydrochloric acid. Laboratories will check the pH of incoming samples to ensure they have been properly preserved. If additional acid must be added to the samples by the lab, they shall notify the Project Chemist or Field Supervisor and the samples must sit for an additional 18 hours before analysis.

Sample preservatives should be identified on the chain of custody (COC).

Solid samples, whether in metal sleeves, wide-mouth glass jars, or other containers, will be labeled and secured appropriately, then placed immediately in an ice chest containing sufficient ice to maintain a temperature range of 6° C through delivery to the laboratory.

Sufficient ice chests and quantities of ice to manage all samples collected during the day (or shift) shall be maintained at the sampling site.

Samples are maintained in ice or, if available, in refrigerators, within a range of 6° C, from the time the sample control manager assumes custody until the samples are packed for shipment and relinquished to the shipper or other transport agent.

All samples are shipped in ice chests packed with sufficient ice to maintain a temperature range of 6° C for at least 24 hours.

One temperature blank shall be placed in the center of each cooler for the laboratory to check the temperature upon arrival at the lab. The temperature blank can be created in the field by pouring deionized water into an empty, unpreserved 40 mililiter VOA or other sample container and should be labeled as a temperature blank and added to the COC to record that it was placed into the cooler.

The receiving laboratory will measure the temperature within the ice chest immediately upon assuming custody of a shipment of samples. This temperature will be noted on the chain-of-custody form. Temperatures in excess of 6° C will be reported immediately to the project chemist. After consultation with the PM, the PC will communicate whether re-sampling is necessary.

Table 1 is a listing of the common analyses with associated containers, preservatives, and holding times. The analyses and associated other data shown in Table 1 give a general background regarding what is required. However, when particular analytical procedures are specified in planning documents, it is best to check directly with the cited method to make sure sample vessels and preservatives are correct.

7.0 REFERENCES

American Public Health Association, 1985. Standard Methods for the Examination of Water and Wastewater, 16th Edition.

40 CFR 136, Code of Federal Regulations, dated July 1, 1990.

United Stated Environmental Protection Agency (USEPA), 1991. Statement of Work for Organics Analysis, Document Number OLMO1.0, USEPA Contract Laboratory Program, June.

United States Environmental Protection Agency (USEPA), 1990a. Statement of Work for Inorganics Analysis, Document Number ILMO1.0, USEPA Contract Laboratory Program, March. November.

EPA (U.S. Environmental Protection Agency), 1990b. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Third Edition, Final Update I, USEPA Office of Solid Waste,

United States Environmental Protection Agency (USEPA), 1982. Methods for Chemical Analysis of Water and Wastes, EPA-600/4-82-055, December

8.0 ATTACHMENTS

Table 1. Sample Containers, Preservation Methods, and Analytical Holding Times

Table 1
Sample Containers, Preservation Methods, and Analytical Holding Times (1 of 2)

Parameter	Matrix	Container	Lid	Preservation	Maximum Holding Times	
					Extraction[a]	Analysis[b]
Metals	Water	500 ml polyethylene	Cap with Teflon® seal	HNO3 to pH<2 (Hg: Ice to [6°C)	-	6 months (Hg: 28 days)
	Soil	4 oz. glass jar	Teflon®-lined lids	none (Hg: Ice to [6°C)	-	6 months (Hg: 28 days)
	Soil	Stainless steel sleeve	Teflon®-lined plastic end-caps	none (Hg: Ice to [6°C)	-	6 months (Hg: 28 days)
Radionuclides	Water	500 ml polyethylene	Cap with Teflon® seal	HNO3 to pH<2	-	6 months
	Soil	4 oz. glass jar	Teflon®-lined lids	none	-	6 months
	Soil	Stainless steel sleeve	Teflon®-lined plastic end-caps	none	-	6 months
Volatiles	Water	40 ml glass vials X 3	Cap with Teflon® septum	HCl to pH<2; Ice to [6°C	-	14 days
	Soil	EnCore sampler X 3	o-ring cap	Ice to [6°C; 48 hours to preserve with methanol or sodium bisulfate	-	14 days
	Soil	Stainless steel sleeve	Teflon®-lined plastic end-caps	Ice to [6°C	-	14 days
Purgeable Hydrocarbons	Water	1 liter glass amber jar	Cap with Teflon® septum	HCl to pH<2; Ice to [6°C	-	14 days
	Soil	EnCore sampler X 3	o-ring cap	Ice to [6°C	-	14 days
	Soil	Stainless steel sleeve	Teflon®-lined plastic end-caps	Ice to [6°C	-	14 days
Extractable Hydrocarbons	Water	1 liter glass amber jar X 2	Teflon®-lined caps	Ice to [6°C	7 days	40 days
	Soil	4 oz. glass jar	Teflon®-lined lids	Ice to [6°C	14 days	40 days
	Soil	Stainless steel sleeve	Teflon®-lined plastic end-caps	Ice to [6°C	14 days	40 days
Total Recoverable Petroleum Hydrocarbons	Water	1 liter glass amber jar X 2	Teflon®-lined caps	H2SO4 to pH<2; Ice to [6°C	-	28 days
	Soil	4 oz. glass jar	Teflon®-lined lids	Ice to [6°C	-	28 days
	Soil	Stainless steel sleeve	Teflon®-lined plastic end-caps	Ice to [6°C		28 days
	Soil (volatiles)	Encore sampler	o-ring cap	Ice to [6°C; methanol within 48 hr	-	14 days
Phenols	Water	1 liter glass amber jar X2	Teflon®-lined caps	Ice to [6°C	7 days	40 days
	Soil	4 oz. glass jar	Teflon®-lined lids	Ice to [6°C	14 days	40 days
	Soil	Stainless steel sleeve	Teflon®-lined plastic end-caps	Ice to [6°C	14 days	40 days
Organochloride Pesticides and PCBs	Water	1 liter glass amber jar X2	Teflon®-lined caps	Ice to [6°C	7 days	40 days
	Soil	4 oz. glass jar	Teflon®-lined lids	Ice to [6°C	14 days	40 days
	Soil	Stainless steel sleeve	Teflon®-lined plastic end-caps	Ice to [6°C	14 days	40 days
Chlorinated Herbicides	Water	1 liter glass amber jar X2	Teflon®-lined caps	Ice to [6°C	7 days	40 days
	Soil	4 oz. glass jar	Teflon®-lined lids	Ice to [6°C	14 days	40 days
	Soil	Stainless steel sleeve	Teflon®-lined plastic end-caps	Ice to [6°C	14 days	40 days
Semivolatiles	Water	1 liter glass amber jar X2	Teflon®-lined caps	Ice to [6°C	7 days	40 days
	Soil	4 oz. glass jar	Teflon®-lined lids	Ice to [6°C	14 days	40 days
	Soil	Stainless steel sleeve	Teflon®-lined plastic end-caps	Ice to [6°C	14 days	40 days

Table 1
Sample Containers, Preservation Methods, and Analytical Holding Times (Page 2 of 2)

					Maximum Holding Times	
Parameter	**Matrix**	**Container**	**Lid**	**Preservation**	**Extraction[a]**	**Analysis[b]**
Dioxins and Furans	Water	1 liter glass amber jar X2	Teflon®-lined caps	Ice to [6°C	28 days	40 days[a]
	Soil	4 oz. glass jar	Teflon®-lined lids	Ice to [6°C	28 days	40 days
	Soil	Stainless steel sleeve	Teflon®-lined plastic end-caps	Ice to [6°C	28 days	40 days
Polynuclear Aromatic Hydrocarbons	Water	1 liter glass amber jar X2	Teflon®-lined caps	Ice to [6°C	7 days	40 days
	Soil	4 oz. glass jar	Teflon®-lined lids	Ice to [6°C	14 days	40 days
	Soil	Stainless steel sleeve	Teflon®-lined plastic end-caps	Ice to [6°C	14 days	40 days
Nitroaromatics and Nitroamines	Water	1 liter glass amber jar X2	Teflon®-lined caps	Ice to [6°C	7 days	40 days
	Soil	4 oz. glass jar	Teflon®-lined lids	Ice to [6°C	14 days	40 days
	Soil	Stainless steel sleeve	Teflon®-lined plastic end-caps	Ice to [6°C	14 days	40 days
Nitroglycerine	Water	1 liter glass amber jar X2	Teflon®-lined caps	Ice to [6°C	7 days	40 days
	Soil	4 oz. glass jar	Teflon®-lined lids	Ice to [6°C	14 days	40 days
	Soil	Stainless steel sleeve	Teflon®-lined plastic end-caps	Ice to [6°C	14 days	40 days
Anions (Cl, nitrate, nitrite, & sulfate)	Water	250 ml polyethylene	Teflon®-lined caps	Ice to [6°C (Cl: none)	-	28 days (NO3 and NO2:48 hrs)
	Soil	4 oz. glass jar	Teflon®-lined lids	Ice to [6°C (Cl: none)	c	28 days (NO3 and NO2:48 hrs)
	Soil	Stainless steel sleeve	Teflon®-lined plastic end-caps	Ice to [6°C (Cl: none)	c	28 days (NO3 and NO2:48 hrs)
Ignitability	Water	250 ml polyethylene	Teflon®-lined caps	none	none	none
	Soil	4 oz. glass jar	Teflon®-lined lids	none	none	none
	Soil	Stainless steel sleeve	Teflon®-lined plastic end-caps	none	none	none
Total Cyanide	Water	1 liter polyethylene	Teflon®-lined caps	NaOH to pH>12; Ice to [6°C	c	14 days
	Soil	4 oz. glass jar	Teflon®-lined lids	Ice to [6°C	c	14 days
	Soil	Stainless steel sleeve	Teflon®-lined plastic end-caps	Ice to [6°C	c	14 days
Hexavalent Chromium	Water	1 liter glass amber jar X2	Teflon®-lined caps	Ice to [6°C	c	24 hours
	Soil	4 oz. glass jar	Teflon®-lined lids	Ice to [6°C	30 days	4 days
	Soil	Stainless steel sleeve	Teflon®-lined plastic end-caps	Ice to [6°C	30 days	4 days
pH	Water	250 ml polyethylene	Teflon®-lined caps	none	-	immediate
	Soil	4 oz. glass jar	Teflon®-lined lids	none	c	immediate
	Soil	Stainless steel sleeve	Teflon®-lined plastic end-caps	none	c	immediate
Field Soil gas	Air or Soil gas	Tedlar bag	None	none	-	3 days
	Air or Soil gas	Summa Canister	None	none	-	14 days

Abbreviations:
ml = milliliter
oz = ounce

a = Starting from the date of collection
b = Starting from the date of extraction; if no extraction is involved, starting from the date of collection
c = Extraction may occur any time prior to analysis. Only the analysis holding time is monitored.